

PATENT SPECIFICATION

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COMPLETE SPECIFICATION

DRAWINGS ATTACHED

Extrusion of Resins

We, MONSANTO CHEMICALS LIMITED, a British Company, of Monsanto House, 10-18, Victoria Street, London, S.W.1., do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the extrusion of thermoplastic synthetic resins, and particularly to an improved process for the production of a foamed thermoplastic synthetic resin by extrusion, as well as to a new die for the extrusion of a foamed resin.

Foamed resins are useful industrial products, because they have excellent heat-insulating properties. They are often made by moulding, but it has been proposed to produce foamed resins by an extrusion process in which a resin in admixture with a gas or vapour is forced under pressure through for example a circular die so that expansion of the gas or vapour causes foaming of the resin. Processes of this type have, however, not been entirely successful for all purposes, and it is for instance difficult to produce foamed polystyrene board by extrusion, because for example the extruded board has a wrinkled surface and is liable to warp.

The process of the invention is one for the production of a foamed thermoplastic synthetic resin by extrusion, and it comprises forming a plurality of separate strands of heat-softened foamable thermoplastic synthetic resin under a pressure sufficient to prevent resin foaming, coalescing the strands while still under pressure and extruding the resulting coalesced mass so as to expand it into a foamed thermoplastic resin.

A preferred process according to the invention comprises extruding a foamable

thermoplastic resin through a die having at its inlet end a plurality of separate channels communicating with a slit at the outlet end of the die, the pressure within the die being sufficient to prevent the resin foaming before it leaves the slit and the relationship between the dimensions of the channels and the dimensions of the slit being such that the streams of resin issuing from the channels spread out to fill the slit and to coalesce whilst still in the unfoamed state.

The invention also comprises a die adapted to the extrusion of a foamed thermoplastic resin, which comprises a block having extending into it at the inlet end of the die a plurality of separate channels communicating with a number of slits in the block at the outlet end of the die, the slits being arranged in the form of a network and each slit having a row of channels associated with it.

The channels in the die used in the preferred process of the invention are normally circular in cross-section, and their other dimension is that along the direction of the flow of the resin, which is the "land" of the channels. However, the channels need not necessarily be circular in cross-section, and they can for instance be of some shape that is substantially circular and that can be said to possess a diameter, for example hexagonal or even perhaps square. Preferably the channels as well as being of circular cross-section are arranged symmetrically behind the slit.

The dimensions of the slit are measured in terms of the land, (which as stated above is the measurement along the direction of the resin flow), and the cross-section, which is made up of the width and the length, the length being the larger of these two linear dimensions. In general the width of

the slit is less than the diameter of channels positioned behind it; for instance the diameter of the channels is often from 1.3 to 10 times, especially from 2 to 5 times, the width of the slit. Although a straight slit is often employed so that the resin is extruded in the form of a board, this is not necessarily so and a curved slit can be used, the curved section of foamed resin thus produced being useful for such purposes as ceiling coving or pipe insulation. Preferably there is more than one slit in the die, for instance a number are arranged parallel to one another or in the form of a network, each slit having its associated row of channels. A network of slits can for example be in the form of a rectangular or triangular grid, for example a series of squares or a six-pointed star within a hexagon. A network of slits can be arranged to produce a hollow product. The length of the slit can be as much as desired, for instance from $\frac{1}{4}$ inch to 60 inches or even more, depending on the dimensions desired in the final extruded foamed resin. The width of the slit also bears some relationship to the thickness of the extruded resin, but is also to some extent dictated by the maximum practical extrusion rate. In general a width of slit between 0.01 and 0.25 inch, for instance from 0.02 to 0.1 inch and especially about 0.025 inch, has been found suitable. The land of the slit can for instance be between 0.05 inch and 2 inches, especially between 0.1 and 0.5 inch.

Preferably the channels and the slit are so designed that the residence time of the resin in the slit is relatively short compared with that in the channels. Such conditions can for example be achieved by suitable choice of the length of "land" of the channels and of the slit. Often, the land of the channels is between 2 and 20 times the land of the slit, and for example where polystyrene is to be extruded the land of the channels can for instance be from 4 to 15 times the land of the slit. Where polyethylene is to be extruded the land of the channels can for instance be from 2 to 6 times the land of the slit. For other resins the ratio can be more or less than these values, depending on whether the viscoelastic memory of the resin is low or high.

The process of the invention is particularly useful for producing foamed resin in the form of a board, whose dimensions depend of course on those of the die and the degree of foaming that occurs when the resin leaves the die. Generally, greater expansion takes place across the width of the slit than along its length, and for instance a board that is 1 or 2 inches thick can be produced from a narrow slit.

The resin is preferably a polymer or copolymer of a vinyl or vinylidene monomer, preferably a hydrocarbon monomer such as for example ethylene, propylene, butadiene, styrene, vinyltoluene or α -methylstyrene, or a substituted monomer such as for example acrylonitrile, vinyl chloride, vinyl acetate, methyl acrylate, methyl methacrylate or ethyl acrylate. Excellent results have been obtained using either polyethylene or polystyrene, and these are the preferred resins. Toughened polystyrene can also be employed.

The resin used in the process is of course foamable, and this means that it is in admixture with a blowing agent which is preferably a normally gaseous substance but can be a volatile liquid. In many cases the blowing agent is one that is normally gaseous but which while under pressure before extrusion is present in the liquid state. Examples of volatile substances that can be used include lower aliphatic hydrocarbons such as ethane, propane, butane or pentane, lower alkyl halides such as methyl chloride, trichloromethane or 1,2-dichlorotetrafluoroethane, and inorganic gases such as carbon dioxide or nitrogen. The lower aliphatic hydrocarbons, especially butane, are preferred. The blowing agent can also be a chemical blowing agent, which can for example be a bicarbonate such as for example sodium bicarbonate or ammonium bicarbonate, or an organic nitrogen compound that yields nitrogen on heating, such as for example dinitrosopentamethylenediamine or barium azodicarboxylate. From 3 to 30% especially from 7 to 20%, by weight based on the weight of the resin is often a suitable proportion of blowing agent, and for example the use of from 7 to 15% by weight of butane in conjunction with polystyrene has given excellent results.

Extrusion dies utilisable in a process according to the invention are exemplified (not to scale) in the Drawings accompanying the provisional specification, in which:

Figure 1 is a front elevation of one construction of die;

Figure 2 is a section along the line II-II in Figure 1;

Figure 3 is a front elevation of a second construction of die;

Figure 4 is a section along the line IV-IV in Figure 3;

Figure 5 is a front elevation of a third construction of die; and

Figure 6 is a section along the line VI-VI in Figure 5.

(The die shown in Figures 5 and 6 is an example of a die according to the invention.)

The die shown in Figures 1 and 2 consists of a mild steel block (1) having extending into it from one side a row of 16

cylindrical channels (2) equally spaced 0.25 inch apart, each channel being 0.825 inch long and 0.04 inch in diameter. The channels communicate with a slit (3) 0.015 inch wide, 4 inches long and having a land of 0.125 inch and thence with the opposite side of the block. The die has means (not shown) for attaching it to the front end of an extruder, so that when the die is in use a foamable resin such as for instance foamable polystyrene is fed into the channels in the direction of the arrow and hence into the slit.

In the die shown in Figures 3 and 4 there are three parallel slits (4, 5 and 6) each slit being similar to that shown in Figures 1 and 2 and having its associated row of channels (7) arranged in a staggered pattern as shown. The slits are spaced 0.25 inch apart.

Figures 5 and 6 show a die having a rectangular network of intercommunicating slits, for example those designated (8), (9), (10) and (11). The slits are all 0.015 inch wide and have a land of 0.125 inch. The three horizontal slits (8) (9) and (10) are each 4 inches long and are spaced 0.25 inch apart, and seventeen vertical slits (11) $\frac{1}{2}$ inch long are spaced 0.25 inch apart along the length of the horizontal slits. Each slit has associated with it a row of channels (12) spaced $\frac{1}{2}$ inch apart of diameter 0.04 inch and land 0.825 inch, the horizontal slits having sixteen channels and the vertical ones two.

The process of the invention is particularly applicable to the production of foamed resins, such as polystyrene, having a density between 0.5 and 10 pounds per cubic foot and especially between 1 and 5 pounds per cubic foot.

The extrusion temperature (that is the temperature of the die and the resin within it) depends to some extent on the softening point of the resin, but in general temperatures between 95°C. and 180°C., preferably between 100°C. and 160°C., are suitable. For example, when foamable polystyrene is being extruded a temperature in the range 130°C. to 160°C. can be used, while for polyethylene somewhat lower temperatures, for instance 100°C. to 120°C., are often very suitable.

The pressure within the die is sufficient to prevent the resin foaming until it leaves the slit, and where the volatile substance is condensable the pressure is preferably greater than the saturated vapour pressure of the volatile substance at the extrusion temperature. Pressures for example greater than 250 pounds per square inch, and especially between 250 and 500 pounds per square inch, can be employed. Preferably the pressure is between 300 and 1000 pounds per square inch.

The process of the invention is illustrated by the following Examples.

EXAMPLE I

This Example describes a process according to the invention for the production of foamed polystyrene.

Foamable polystyrene pellets containing 7% by weight of butane were extruded at a pressure of 500 pounds per square inch and temperature 150°C. through the die described above and illustrated in Figures 5 and 6, at a flow rate of 30 pounds per hour, the extruded polystyrene being passed between a pair of rollers set $\frac{1}{4}$ inch apart.

There was produced a board of foamed polystyrene 6 inches wide by $\frac{1}{2}$ inch thick having a density of $1\frac{1}{2}$ pounds per cubic foot. It was of good strength in both the transverse and longitudinal directions, and although the effect of the channels was discernable in the board the surfaces were satisfactorily smooth.

For the purposes of comparison a similar foamable polystyrene mixture was extruded through a die comprising a number of channels without any slit, and attempts were made to consolidate the resulting foamed strands by passing them between rollers or by passing them through a restricted shaping box. The results were completely unsatisfactory in each instance because of the lines of weakness which existed along the joins between the strands. Attempts to extrude foamed polystyrene through slit dies (without any channels in communication with them) also resulted in an unsatisfactory product, the board being badly wrinkled and warped.

EXAMPLE II

This Example describes a process according to the invention for the production of foamed polyethylene.

Foamable polyethylene containing 15% by weight of butane and 3% of finely divided silica (to act as nucleating agent) was extruded at a pressure of 400 pounds per square inch and temperature 110°C. through a die similar to that described above and illustrated in Figures 5 and 6, but having three horizontal and 3 vertical equally spaced slits, each slit having a length of 0.4 inch, a width 0.025 inch and a land 0.125 inch, and each having communicating with it 2 channels of diameter 0.11 inch and land 0.5 inch. The flow rate was 10 pounds of resin per hour.

There was produced a hollow foamed polyethylene section similar in shape to the cross-section of the die, but having external dimensions approximately 1 inch square and having a density of 3 pounds per cubic foot. It was resilient but of good strength in both the transverse and longitudinal directions, and it was difficult

to discern the effect of the channels in the foamed product.

Comparative experiments similar to those described in Example 1 failed to produce a useful product, for the reasons outlined in that Example.

WHAT WE CLAIM IS:—

1. A process for the production of a foamed thermoplastic synthetic resin by extrusion, which comprises forming a plurality of separate strands of heat-softened foamable thermoplastic synthetic resin under a pressure sufficient to prevent the resin foaming, coalescing the strands while still under pressure and extruding the resulting coalesced mass so as to expand it into a foamed thermoplastic resin.

2. A process according to Claim 1, in which the resin is polystyrene.

3. A process according to Claim 1, in which the resin is polyethylene.

4. A process according to any of Claims 1 to 3, which comprises extruding the foamable thermoplastic synthetic resin through a die having at its inlet end a plurality of separate channels communicating with a slit at the outlet end of the die, the pressure within the die being sufficient to prevent the resin foaming before it leaves the slit and the relationship between the dimensions of the channels and the dimensions of the slit being such that the streams of resin issuing from the channels spread out to fill the slit and to coalesce whilst still in the unfoamed state.

5. A process according to Claim 4, in which the channels of the die are of substantially circular cross-section and are arranged symmetrically behind the slit.

6. A process according to either of Claims 4 and 5, in which the diameter of the channels is from 2 to 5 times the width of the slit.

7. A process according to any of Claims 4 to 6, in which the residence time of the resin in the slit is shorter than the residence time in the channels.

8. A process according to any of Claims 4 to 7, in which in the die there are a number of slits arranged in the form of a network, each slit having an associated row of channels.

9. A process according to any of Claims 4 to 8, in which the width of the slit is from 0.02 to 0.1 inch.

10. A process according to any of Claims 4 to 9, in which the land of the slit is between 0.1 inch and 0.5 inch.

11. A process according to any of Claims 4 to 10, in which the resin is poly-

styrene and the land of the channels is from 4 to 15 times the land of the slit.

12. A process according to any of Claims 4 to 10, in which the resin is polyethylene and the land of the channels is from 2 to 6 times the land of the slit.

13. A process according to any of the preceding claims, in which there is used a die substantially as hereinbefore described and illustrated with reference to the Drawings accompanying the provisional specification.

14. A process according to Claim 1 substantially as described in either of the Examples.

15. A foamed thermoplastic synthetic resin that has been produced by a process according to any of Claims 1 to 3.

16. A foamed thermoplastic synthetic resin that has been produced by a process according to any of Claims 4 to 14.

17. A die adapted to the extrusion of a foamed thermoplastic resin in a process according to Claim 4, which comprises a block having extending into it at the inlet end of the die a plurality of separate channels communicating with a number of slits in the block at the outlet end of the die, the slits being arranged in the form of a network and each slit having a row of channels associated with it.

18. A die according to Claim 17, in which the channels are of substantially circular cross-section and are arranged symmetrically behind the slits.

19. A die according to either of Claims 17 and 18, in which the diameter of the channels associated with a slit is from 2 to 5 times the width of the slit.

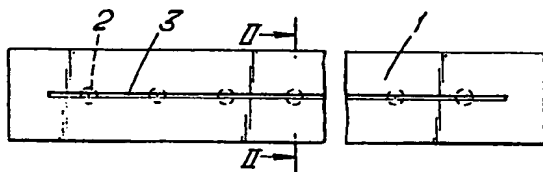
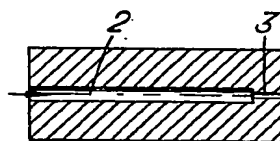
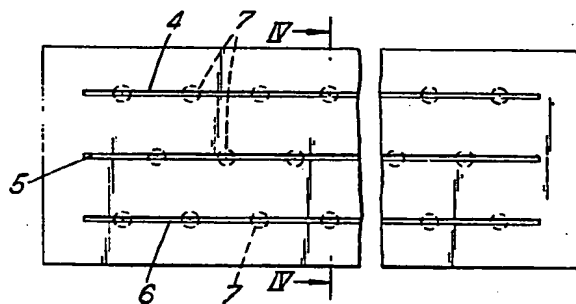
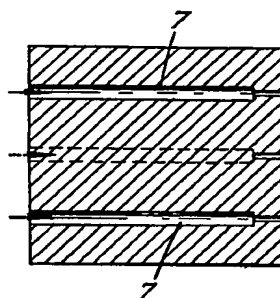
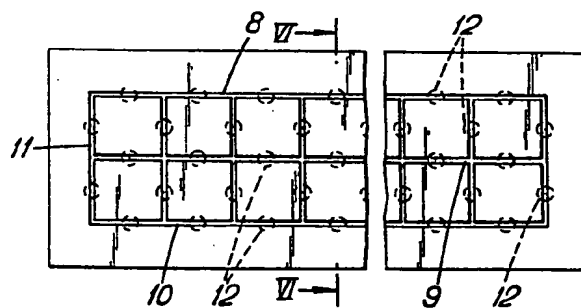
20. A die according to any of Claims 17 to 19, in which the dimensions are as defined in any of Claims 9 to 12.

21. A die adapted to the extrusion of a foamed thermoplastic resin, substantially as hereinbefore described and illustrated with reference to Figures 5 and 6 of the Drawings accompanying the provisional specification.

22. A process according to Claim 4, in which there is used a die according to any of Claims 17 to 21.

23. A foamed thermoplastic synthetic resin that has been produced by a process according to Claim 22.

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Fig. 1.*Fig. 2.**Fig. 3.**Fig. 4.**Fig. 5.**Fig. 6.*